

Testing for Pb in Painted Children's Products Tube-based Portable XRF Solutions

Collaboration with CPSC

Innov-X Systems, Inc.
Woburn, MA

Friday, November 7th, 2008
CPSC Public Meeting:
X-ray Fluorescence (XRF) Testing for Lead in Paint

Historical Portable XRF Solutions in Testing for **Pb**

Innov-X Tube-based Portable XRF has demonstrated success in the following **Regulatory Driven Pb Testing**:

- **HUD-PCS Approved:** **Pb** in Residential Paint
- **Meets RCRA Metals:** **Pb** in soil, liquids and filters
- **Meets EPA Method 6200:** **Pb** in soil
- **Meets NIOSH Method 7702:** **Pb** in air filters
- **Meets OSHA Method ID-204:** **Pb** in air filters and dust wipes
- **World Wide Acceptance for RoHS Screening:** **Pb** in Consumer Electronic Products (plastics, alloys, mixed materials)
- **Standard FDA & Border Security Inspection Tool:** **Pb** in food, wrappers, containers, toys, trinkets, jewelry, other imported consumer products



Portable XRF *Pb* Testing Methods

- Screening for the presence of Pb
 - LOD-dependant; between 2-15ppm depending on matrix (polymer, alloy, mixed) and power of x-ray tube
- Pass/Fail tests for preset levels of Pb
 - Pre-programmed calibrations using known Pb standards and programmed pass/fail levels (thresholds)
- Semi-quantitative analysis of Pb
 - Pre-programmed calibrations using known Pb standards with matching matrices
- Quantitative analysis of Pb
 - Sample preparation of Pb standards and unknowns are the same; completely homogenous with flat surfaces



The screenshot shows the 'Innov-X Results' window. It displays 'Reading #26 23-Aug-2006' and 'RoHS/WEEE Mode'. The results are categorized as 'Mixed' and 'Fail'. A table lists elements and their concentrations in ppm and percentage, along with a pass/fail status. At the bottom, there are buttons for 'Back', 'Next', and 'Exit', and a menu bar with 'File', 'Edit', and 'View'.

Element	ppm / %	+/-	Result
Cr	< 2 ppm		Pass
Hg	< 260 ppm		Pass
Cd	< 487 ppm		Pass
Br	6.32 %	0.46 %	Fail
Pb	4554 ppm	381 ppm	Fail

Portable XRF Configurations for **Pb** Testing

• Closed beam portable systems (X-50)



- High power **10W** (50kV-200 μ A) for lowest detection limits (2-7ppm for *Pb*)
- Closed beam (radiation enclosure) for situations that require and/or are more comfortable with closed beam systems for higher kV radiation
 - On low density samples like plastics, there is more *scattered* radiation from a 50kV source. In air, higher-energy x-rays travel further, thus creating an exposure risk further from the XRF. Hence, 50kV operators may prefer the radiation enclosure of the X-50.

Portable XRF Configurations for **Pb** Testing

Open beam handheld systems (Alpha/Omega/Import Guard)



4W (40kV-100 μ A) with SmartFiltering
tunes the x-ray emissions to match
to elements to deliver optimal
performance across the periodic
table (***10-15ppm for Pb***)

- **Other Operator Safety Considerations:**
“In air” sensor & shutdown; “deadman” or
touch trigger; two-handed operation if
needed. Use of metal snout for heat
dissipation – not hot to the touch - and
electromagnetic shielding for interference
free 2-way emergency radio
communication.
 - » Note: Handheld systems can be used
in test stands for enclosed operation,
but are no longer “in-situ”
measurements.

A Complex Problem

Checking for toxic metals in imported toys, jewelry, and other consumer items



Perform On-the-Spot Screening



Test right through the packaging – no need to destroy goods to get results.

Use Portable XRF to test:

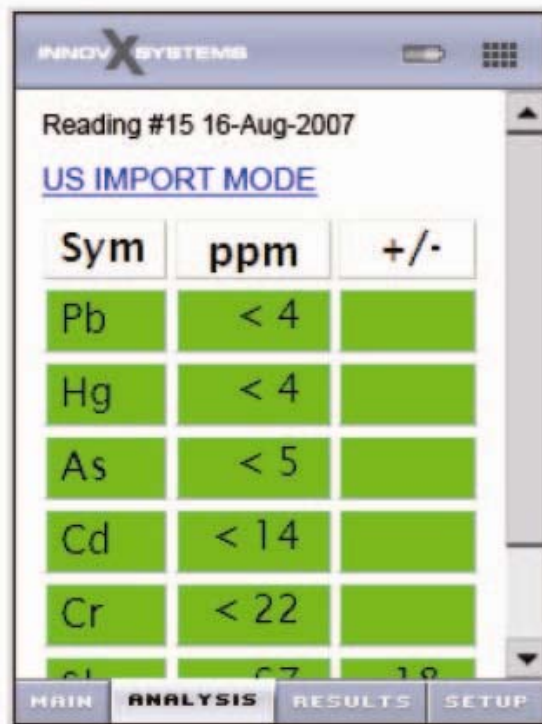


on the manufacturing floor



in the warehouse

Simple Display – PASS/FAIL



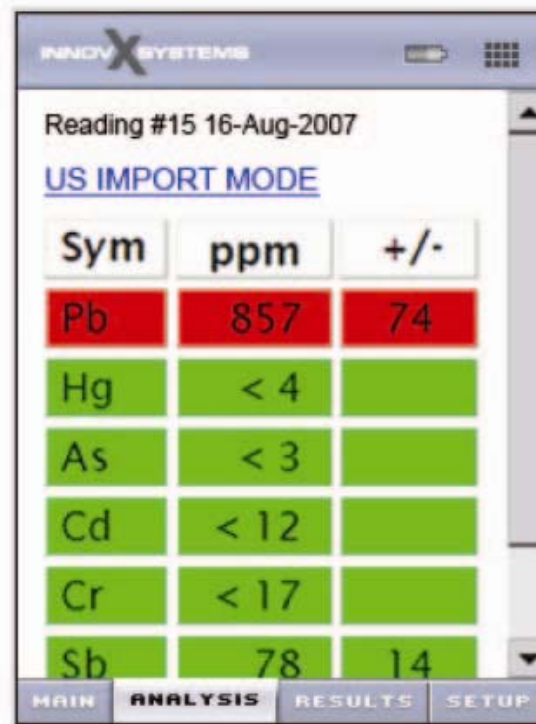
Reading #15 16-Aug-2007

US IMPORT MODE

Sym	ppm	+/-
Pb	< 4	
Hg	< 4	
As	< 5	
Cd	< 14	
Cr	< 22	
Cu	< 67	18

MAIN ANALYSIS RESULTS SETUP

Sample Pass Screen



Reading #15 16-Aug-2007

US IMPORT MODE

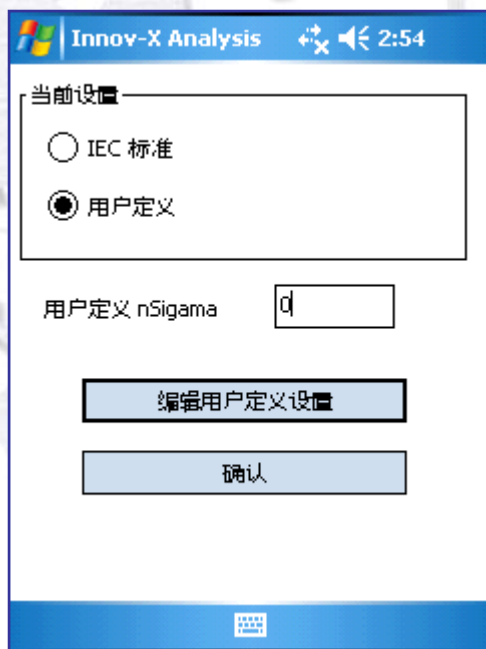
Sym	ppm	+/-
Pb	857	74
Hg	< 4	
As	< 3	
Cd	< 12	
Cr	< 17	
Sb	78	14

MAIN ANALYSIS RESULTS SETUP

Sample Fail Screen



Multi-language Software for Global Business



- Chinese
- Simplified
- Traditional
- Japanese
- Spanish
- French
- German
- Russian

Hong Kong, China
Asia Pacific HQ

Technical Details - Outline

- XRF Calibrations
- How to test toys for Consumer Products Safety Commission (CPSC) levels
- Screening, Quantitative Analysis options
- Small spot System Options



XRF Calibration:

Painted or Layered Samples

Example: lead paint on a substrate.

XRF results in units of $\mu\text{g}/\text{cm}^2$

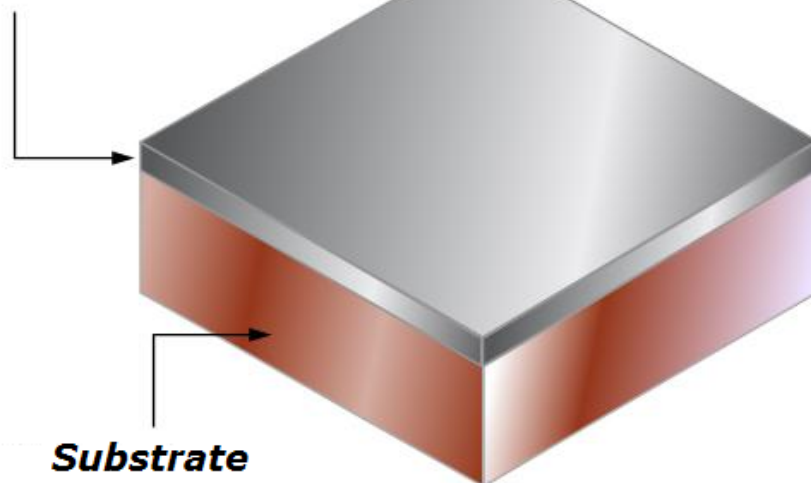
CPSC regulations requires < 600 ppm in homogeneous samples.

This means paint layers treated separately from substrate.

No direct comparison between XRF results and CPSC level.

Paint Layer

Pb must be < 600 ppm in the paint (SPSC)



Substrate

Pb must be < 600 ppm here also

XRF Calibration:

Bulk samples

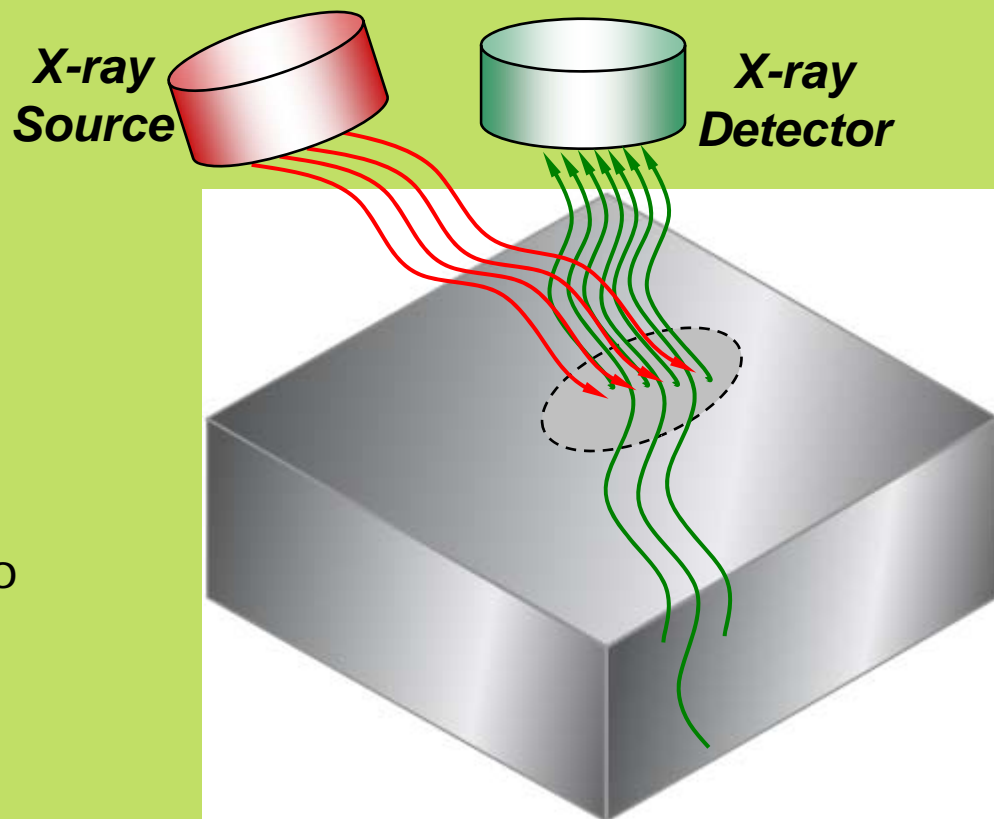
Examples: Soils, liquids, alloys – thick, uniform sample types

XRF results in units of ppm

CPSC regulations requires < 600 ppm in homogeneous samples

Direct comparison of XRF result to CPSC level -> PASS/FAIL

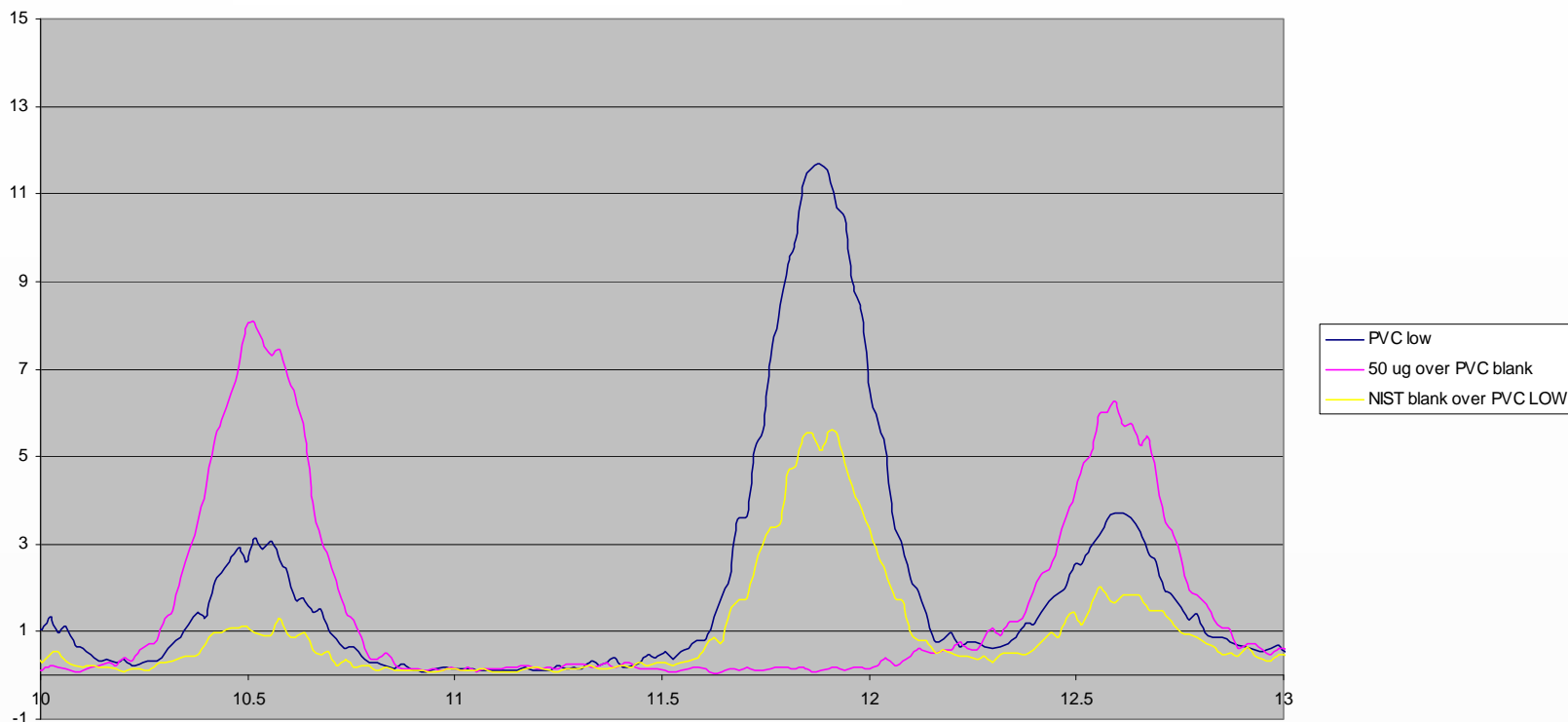
However most toy products don't fit this physical form



Portable XRF Data Quality

400ppm Pb in PVC: Statistical difference between surface lead and bulk lead.

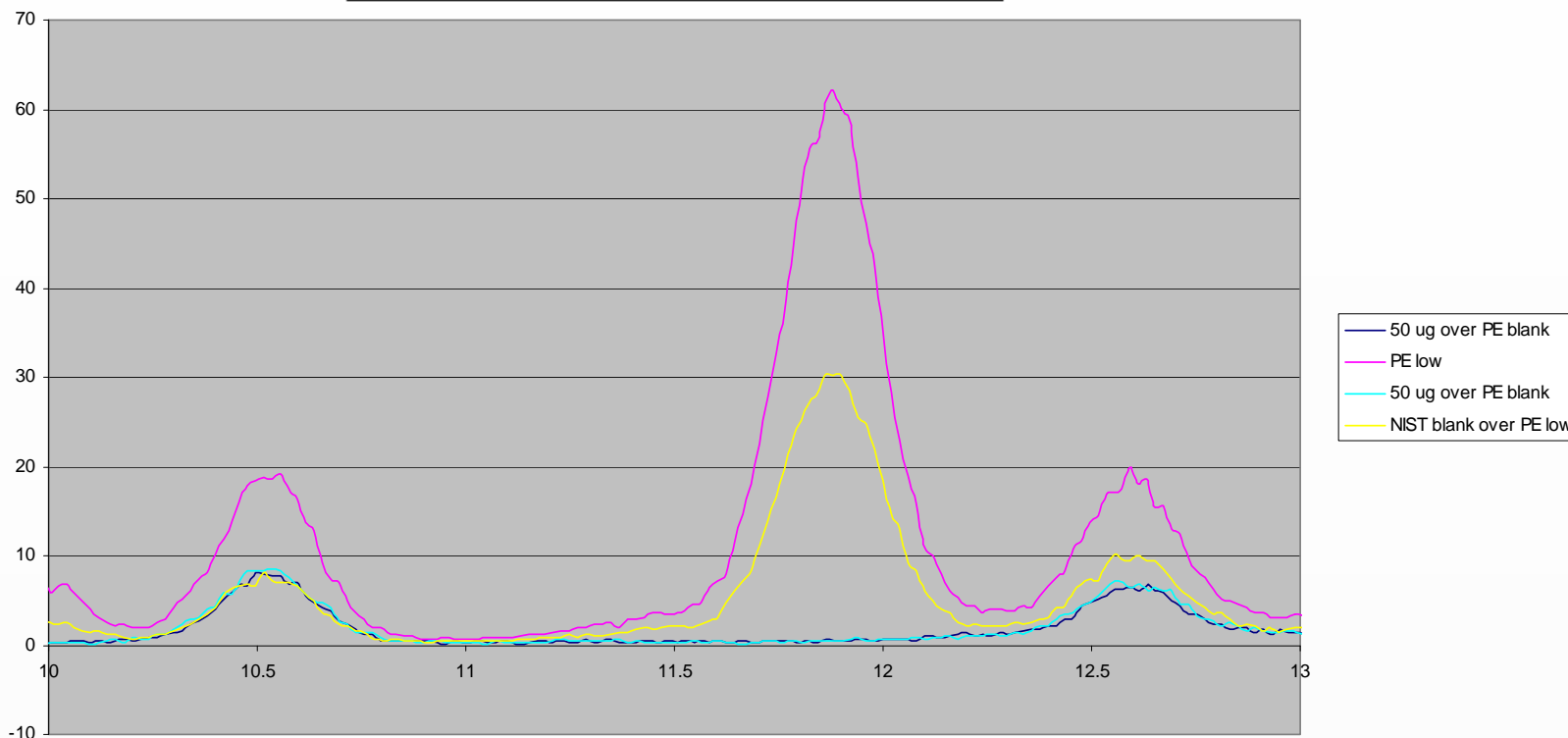
	PVC low	50ug over PVC blank	NIST blank over PVC
10.4--10.3	28.32	78.17	11.19
12.5-12.7	33.93	58.42	18.18
a/t ratio	0.83	1.30	0.62



Portable XRF Data Quality

400ppm Pb in PE: Statistical difference between surface lead and bulk lead.

	PE low	50ug over PE blank	NIST blank over PE low
10.4--10.6	182.12	75.27	73.18
12.5-12.7	178.78	83.77	96.40
a/b ratio	1.02	1.18	0.76

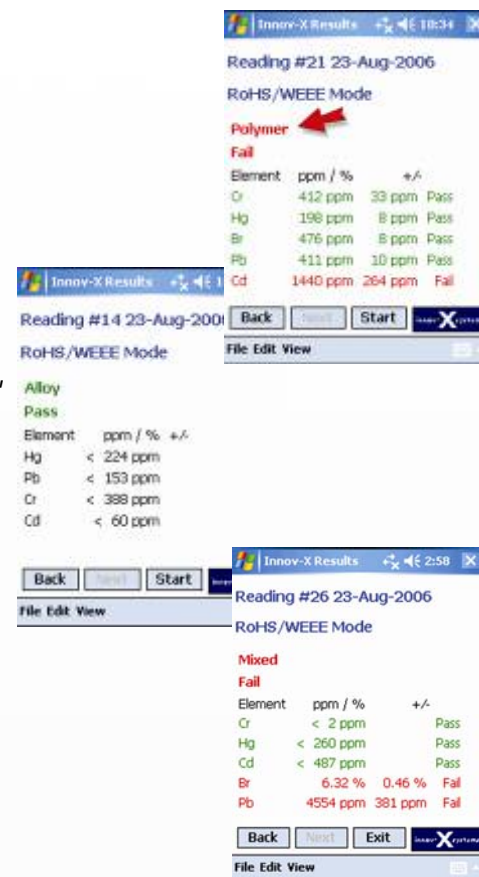


Sample Identification Challenge: Polymer, metal or “mixed”?

Identifying if a bulk sample is a polymer, metal or ‘mixed’ is critical in determining the correct source settings and calibration method for successful analysis. These common sources of operator error are automatically eliminated with the Innov-X patented method ⁽¹⁾.

Innov-X Portable XRF analyzers automatically recognize a bulk sample as a polymer, an alloy, or “mixed”, meaning non-homogeneous. The analyzer then automatically selects the correct filter and tube settings for optimal analysis of that sample. Finally, and most importantly, it also automatically selects the correct calibrations and FP adjustments (polymer type matrix versus alloy) for accurate analysis.

Hence, the Innov-X Smart Technology means the user does not have to manually select the matrix type, tube or filter settings, calibrations or FP adjustments for each sample. This substantially reduces operator error if the wrong sample type is selected or if the operator forgets to change the sample type. Our extensive experience with method developments for the numerous challenges of RoHS testing which includes samples that are plastic (casings), metal/alloy (solders) and “mixed” or non-homogeneous (PCB) - being tested by multi-lingual, non-scientists partially led to the development of this patented method ⁽¹⁾.



(1): US Patent# 7,430,274

Introducing the C^3

C^3 = Camera + Collimation + Coatings

- The C^3 incorporates input from the major testing labs, RoHS consultants and large user groups.
- There are five critical features:
 1. Excellent optics – no parallax
 2. Multiple beam sizes: narrow and full-field.
 3. Portability & Image Capture without a PC
 4. Analyze bulk materials and coatings.
 5. Automatically recognizes sample type (polymer, alloy or mixed) for error-free operation.

Innov-X Co-linear Optics – No Parallax

- Image light (**red**) and x-rays travel same pathway through collimator, to sample
- Eliminates parallax (image location and x-ray location on different spots of sample). Eliminates need for laser guide (safer).

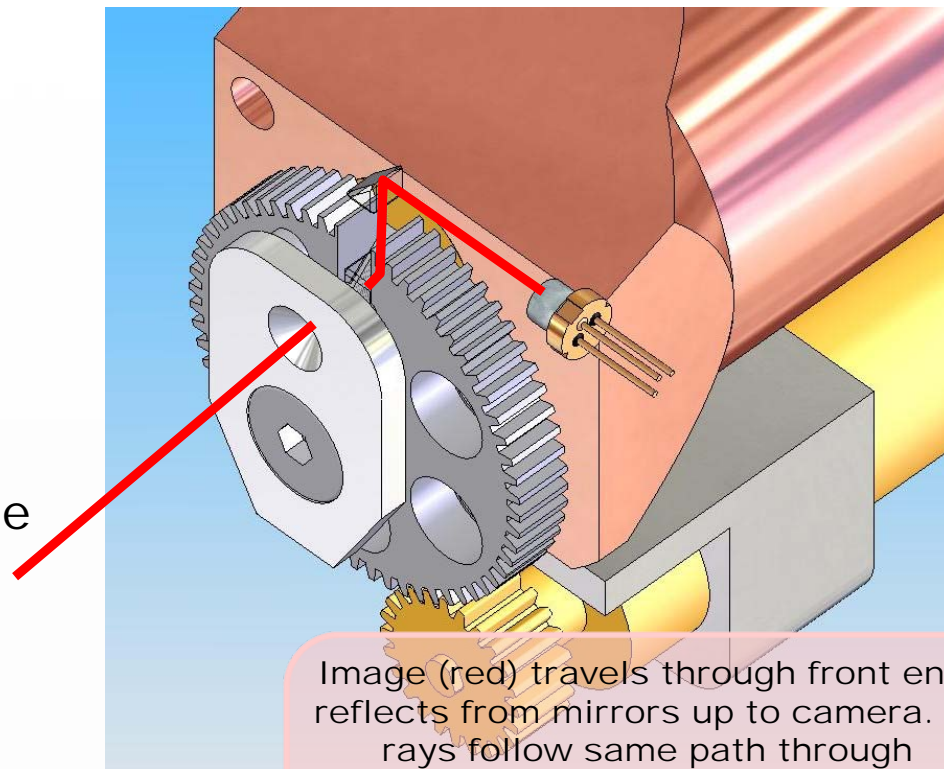


Image (red) travels through front end, reflects from mirrors up to camera. X-rays follow same path through collimator and front end to strike sample in same location as image. This yields the most precise alignment of x-rays on target.

The Problem with Parallax



Parallax occurs with optics designs that are not collinear. When the sample is moved away slightly from the front of the analyzer, the area being imaged is located differently than the sample region being x-rayed. The XRF result does not correspond with the photo of the area being tested for compliance, leading to analysis errors. The elimination of parallax was cited as a critical feature by our RoHS user community, due to the likelihood of operator error.



C³ Portability & Image Capture

- View image on analyzer.
- Photo-documentation: Test results are displayed and stored with image
- Analyzer does not require a PC to work.



Why a Choice in Beam Size?

- XRF precision and limit of detection (LOD) is proportional to the square of the beam diameter.
- Example: for same testing time, LOD is $[12/3]^2$ or 16 times lower for a 12 mm versus a 3 mm beam size.
- OR, for the same LOD, the testing time is 16 times faster!
- In practice, tube currents are increased to mitigate this effect but factors of 3X to 5X are typical.
- A single, smaller beam size also isn't ideal for conducting high-throughput screening or fast QC checks on homogenous samples; longer test times are needed, compared to a 12 mm spot, to achieve the detection limits needed for compliance.

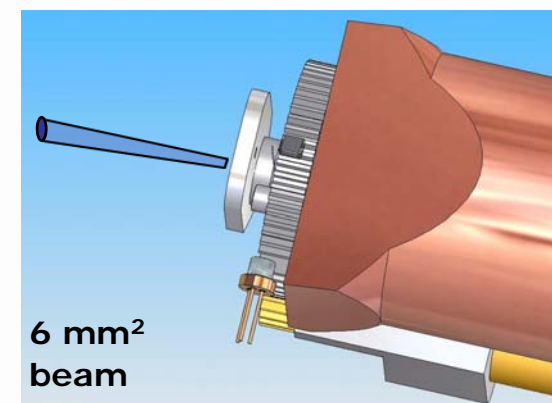
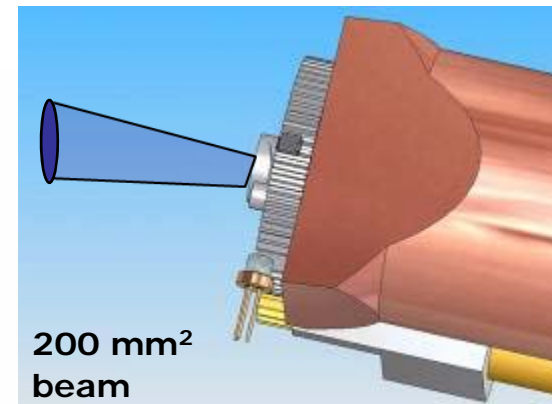
The C³ Offers Multiple Beam Spot Sizes

Full beam size (12 mm, 200 mm²) for large samples or rapid screening.

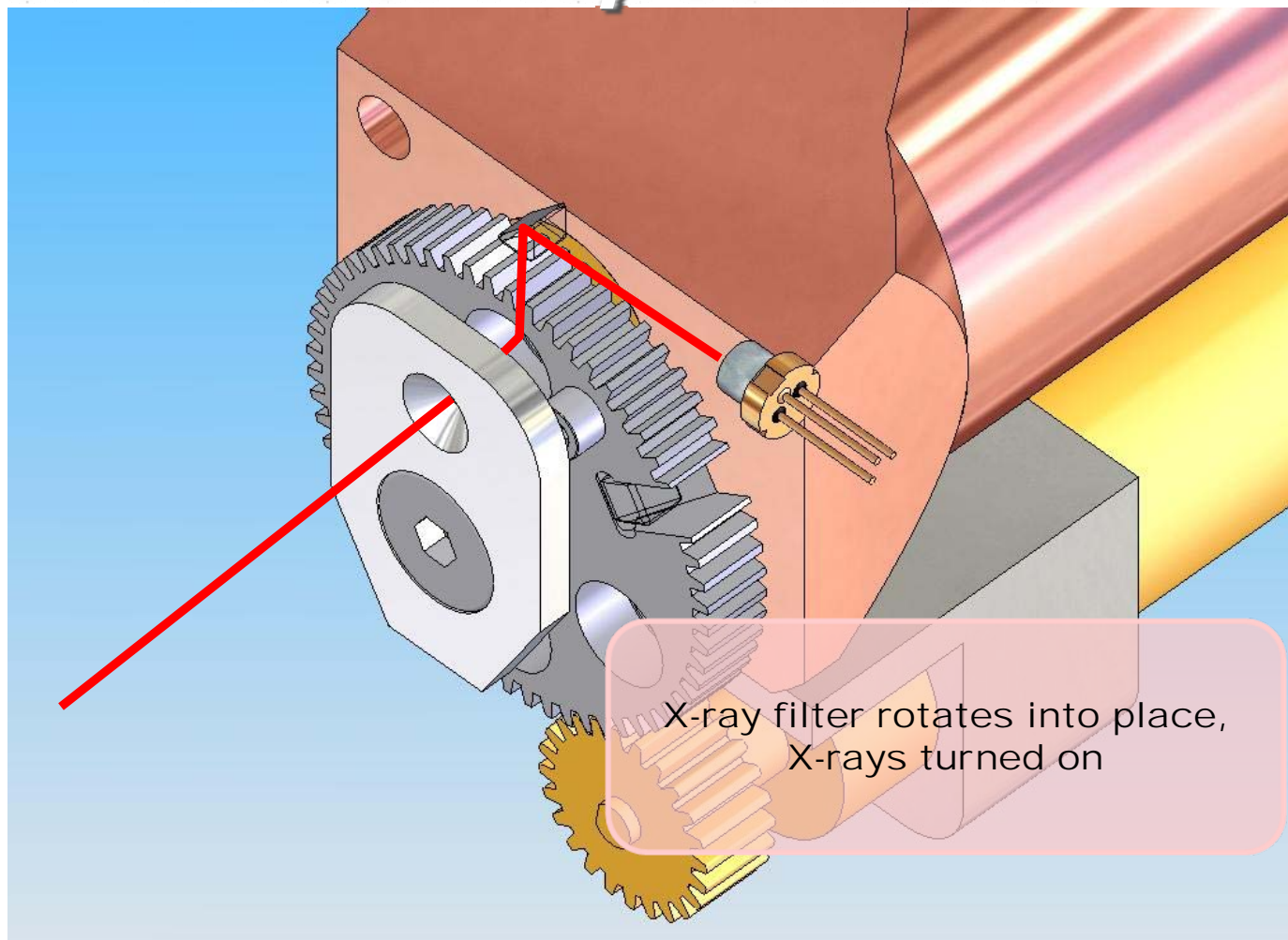
- Some RoHS samples REQUIRE full beam size, examples:
- Measuring < 1,000 ppm Pb in a Sn-based solder.
- Measuring < 100 ppm Cd in a dense alloy

Collimated sizes 2 mm or 3 mm (6 mm²) diameter for components testing

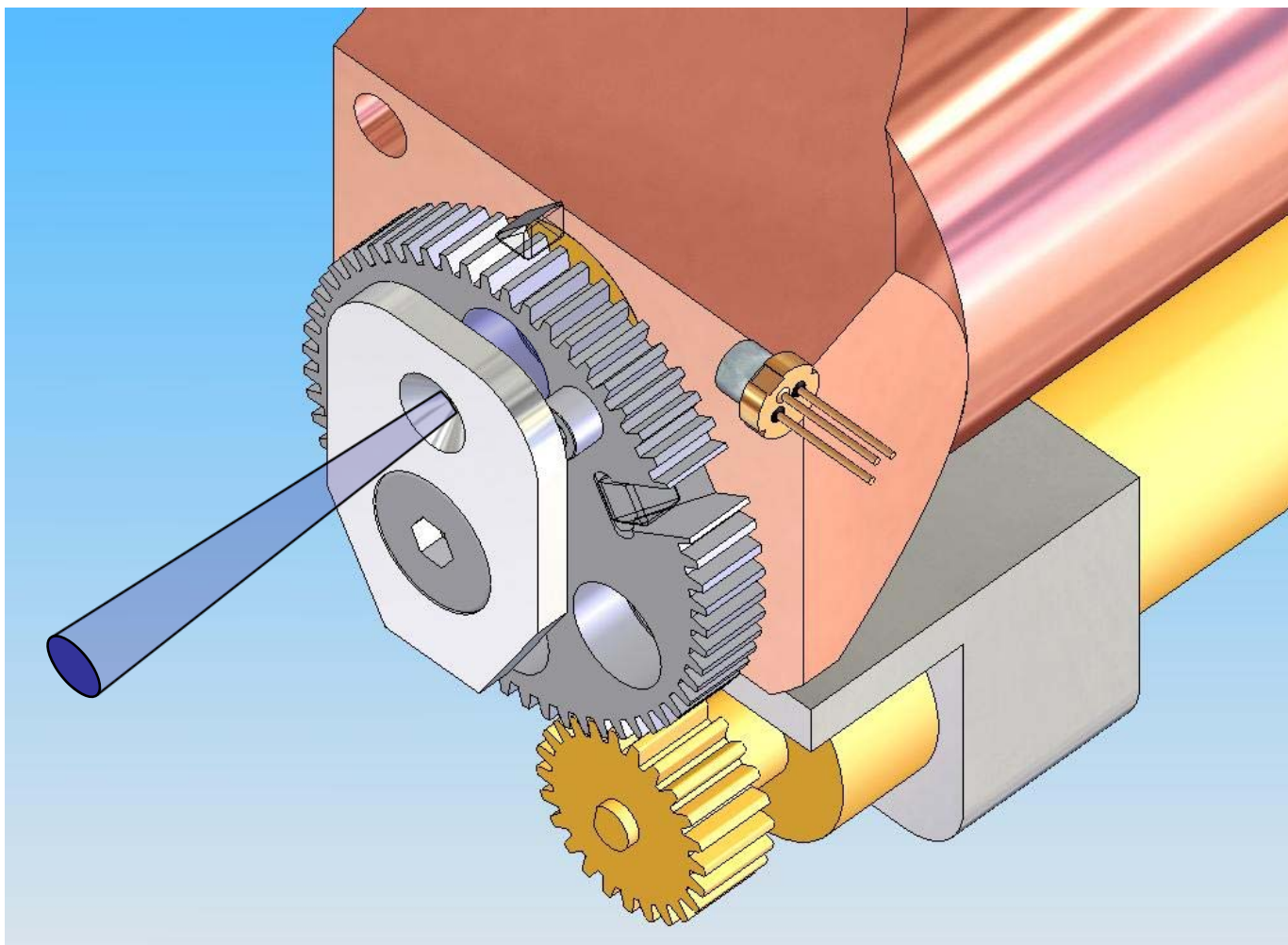
- Toggle between full beam size and small beam size easily



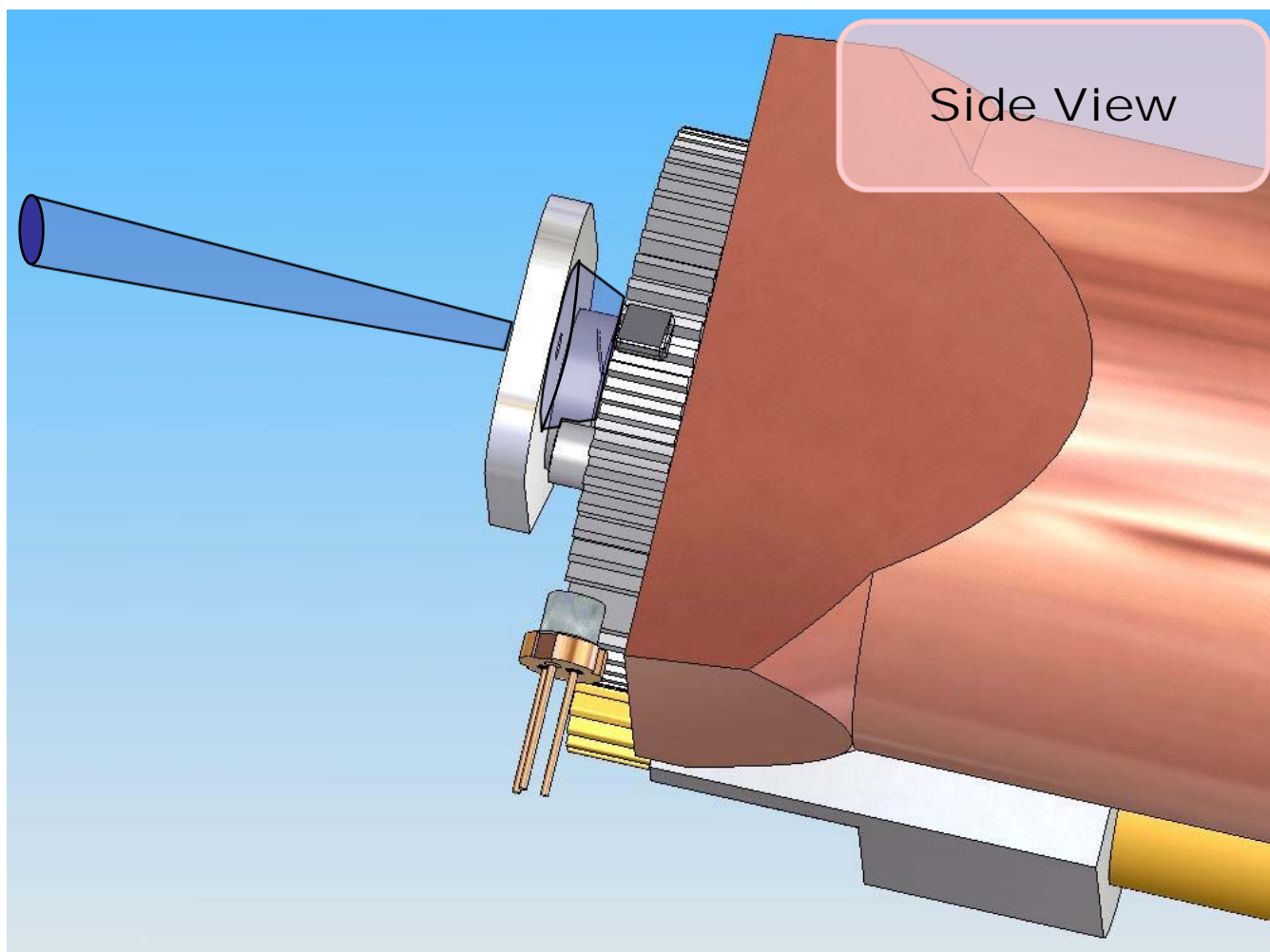
For X-ray Analysis: Filter Wheel Repositions



X-Ray Passes Through Collimator



X-Ray Passes Through Collimator



XRF Test Performed Automatically

Test results are displayed, and stored with image



INNOV-X SYSTEMS		
Reading #15 16-Aug-2007		
<u>US IMPORT MODE</u>		
Sym	ppm	+/-
Pb	< 4	
Hg	< 4	
As	< 5	
Cd	< 14	
Cr	< 22	
Cu	67	18
MAIN ANALYSIS RESULTS SETUP		

Sample Pass Screen

INNOV-X SYSTEMS		
Reading #15 16-Aug-2007		
<u>US IMPORT MODE</u>		
Sym	ppm	+/-
Pb	857	74
Hg	< 4	
As	< 3	
Cd	< 12	
Cr	< 17	
Sb	78	14
MAIN ANALYSIS RESULTS SETUP		

Sample Fail Screen

Portable XRF Solutions for a Complex Problem

Helping to Protect Consumers from Lead Exposure

